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Anatase TiO_2 co-doped with silver and ceria for antibacterial application

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ABSTRACT

Anatase TiO_2 co–doped with Ag and CeO_2 (Ag/CeO_2–TiO_2) was prepared by the peroxo sol-gel method for antibacterial application. The as-prepared materials were characterised by high resolution transmission electron microscopy (HRTEM), X-ray diffraction (XRD), scanning electron microscopy (SEM) and X-ray photoelectron spectroscopy (XPS). The antibacterial activities of the samples were tested against two different bacteria: *Escherichia coli* (OP50), a Gram-negative organism and *Staphylococcus aureus* (USA300), a Gram-positive organism. The antibacterial effectiveness of the Ag/CeO_2–TiO_2 coating was > 99.99%, i.e. it was extremely effective against both *E. coli* (OP50) and *S. aureus* (USA300) after either 30 min of illumination with UVA radiation or 24-h of incubation in the dark. The Ag/CeO_2–TiO_2 coating was found to be significantly more active than pure TiO_2 and TiO_2 doped with CeO_2 or Ag alone. Therefore, Ag/CeO_2–TiO_2 can be used as a coating material for the disinfection of both Gram-positive and Gram-negative bacteria.

1. Introduction

Over the past several years, titanium dioxide (TiO_2) has received significant attention and has become the most promising photocatalyst for numerous applications because of its relatively low cost, low toxicity, high stability and high efficiency [1,2]. UV/TiO_2 disinfection is one of the most effective technologies in the field of water treatment, especially the removal of faecal coliforms (e.g. *Escherichia coli*) in drinking water [3].

Rare earth elements, in particular cerium, have been used as antimicrobial agents in biological dressings for a long time due to their advantages of high safety and a broad spectrum of antibacterial activity [4]. The toxicity of TiO_2 nanoparticles, as well as CeO_2 nanoparticles, is due to the oxidative stress or/and production of reactive oxygen species (ROS), including the superoxide ion (O_2^{-}), hydroxyl radical ($OH\cdot$), hydrogen peroxide (H_2O_2) and singlet oxygen, under UV irradiation. The generated ROS can cause cell membrane damage, cessation of the cell cycle, DNA damage and lipid peroxidation of microorganisms via direct contact between cells and nanoparticles, which leads to cell death [5–7]. In addition, doping with CeO_2 can promote the formation of oxygen vacancies and the relatively high mobility of ROS because of its ability to shift between CeO_2 and Ce_2O_3 under redox reaction, which improves its photocatalytic application against bacteria [4,8,9]. However, the activity of CeO_2–TiO_2 mixed oxide is limited to conditions of irradiation; in other words, the antimicrobial properties of the materials are inactive in the dark [7]. Thus, doping with a suitable noble metal such as silver (Ag) can be used to enhance antibacterial activity under both UV exposure and in the dark [10,11]. Silver nanoparticles (AgNPs) have received much attention as antimicrobial materials, and they are active even in the absence of light [11,12]. A smaller AgNP size results in higher specific surface area, which can be easily oxidised to release the antimicrobial Ag ions into an aqueous environment. Consequently, the Ag ions readily interact with the organic compounds of microorganisms [13–15].

In previous studies [16,17], Ag-doped TiO_2 (Ag/TiO_2) and CeO_2-doped TiO_2 (CeO_2–TiO_2) sols were successfully prepared by the peroxo sol-gel method [18,19]. The as-prepared sol was neutral, stable and transparent, showing enhanced photocatalytic degradation of methylene blue aqueous solution. This method possesses many advantages; for example, (i) further high-temperature treatment to obtain the anatase structure of TiO_2 is not required, and (ii) the use of H_2O_2 as an oxidising agent in the process results in a neutral and well-dispersed colloidal suspension, which is easier to apply to various kinds of substrate without facing corrosion problem.

Therefore, in this study, the peroxo sol-gel method was used to prepare Ag and CeO_2 co-doped TiO_2 sol as a coating material for application as an antibacterial agent. Since in earlier studies of Ag/TiO_2 and CeO_2–TiO_2 [16,17], the antibacterial properties of such materials were not reported, Ag/TiO_2 and CeO_2–TiO_2 were also prepared for comparison of their antibacterial activities.

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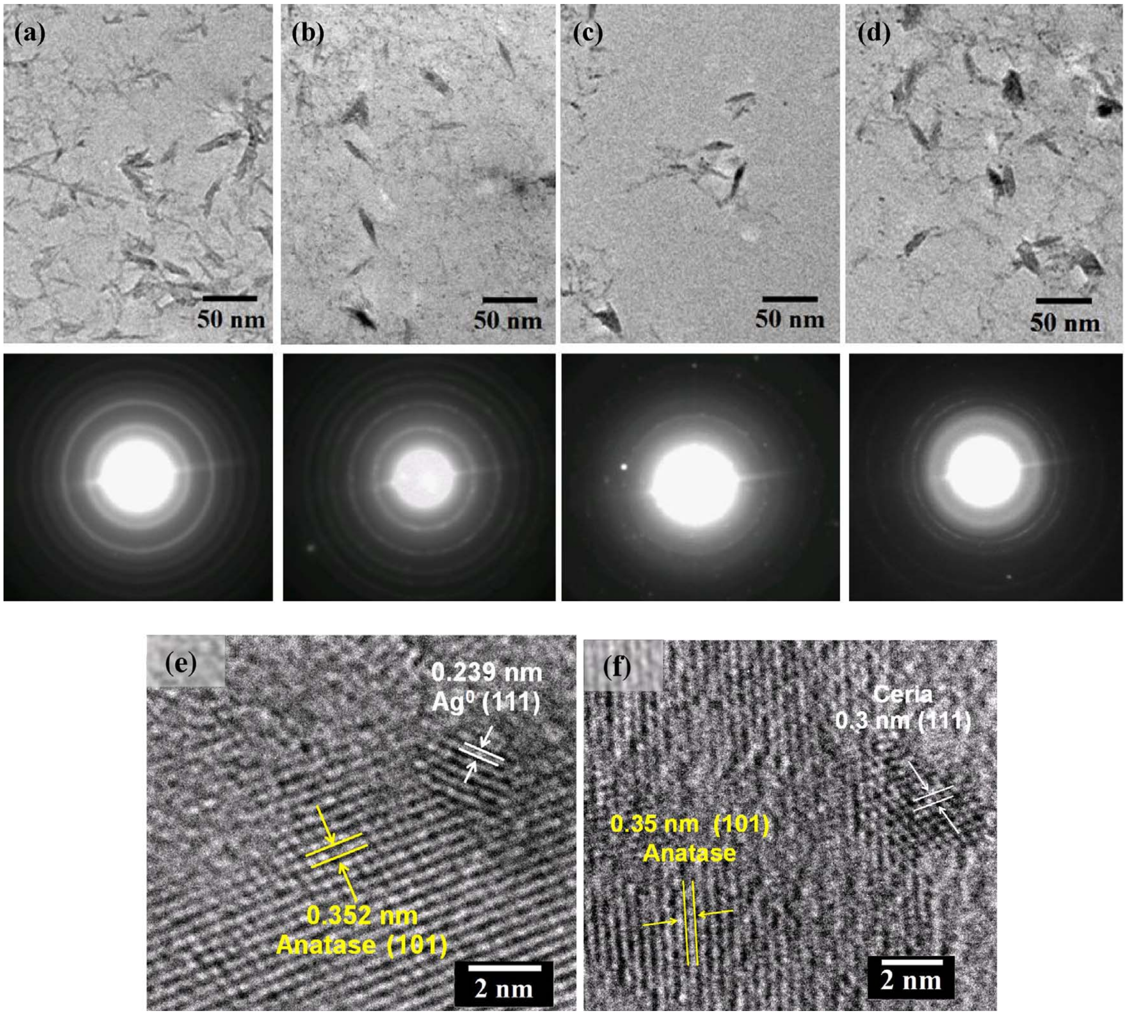

Fig. 1. TEM images (top) and selected area electron diffraction (SAED) pattern (bottom) of (a) TiO_2, (b) CeO_2–TiO_2, (c) Ag/TiO_2 and (d) Ag/CeO_2–TiO_2, (e) and (f) HRTEM micrographs of Ag/CeO_2–TiO_2, representing the lattice spacing attributed to the (101) plane of titania, the (111) plane of ceria and the (111) plane of AgNP.

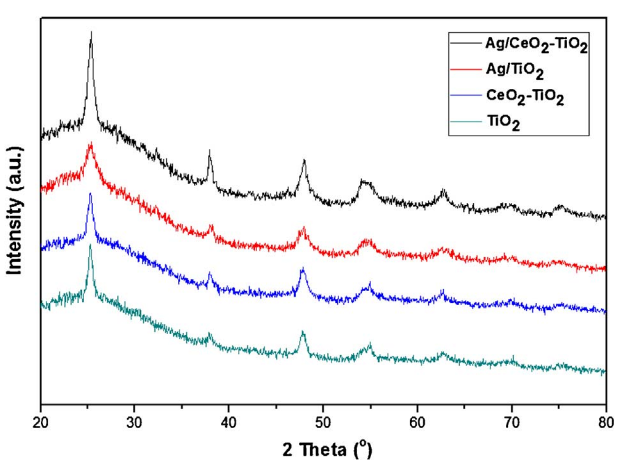

Fig. 2. XRD patterns of dry powders obtained from the sols.

2. Experimental section

2.1. Materials

Titanium tetrachloride (purity > 99.9%) and hydrogen peroxide (35% in water) were purchased from Showa. NH_4OH was purchased from Merck. Cerium oxide (aqueous nanoparticle dispersion, < 5 nm (DLS), 20% solids by weight, pH ~8) was purchased from Sigma-Aldrich. Silver nitrate ($AgNO_3$) was obtained from Showa Chemical Co. Distilled water was used throughout the experiments.

2.2. Synthesis of TiO_2, and Ag and CeO_2 co-doped TiO_2 sol

The typical procedure for preparing TiO_2 sol is as follows: 2.94 ml $TiCl_4$ was slowly added dropwise into 150 ml 1 M HCl aqueous solution under magnetic stirring; it was kept in an ice bath to maintain the temperature at 0 °C. An aqueous solution of 1 M NH_4OH was then added dropwise to form a white hydrated titanium oxide gel, $Ti(OH)_4$. The pH value of the solution was adjusted to 8.0 by adding adequate amounts of NH_4OH solution. After stirring for 30 min, the $Ti(OH)_4$ gel was filtered and washed with distilled water several times until no chloride ions were detected by chloride test strips (QUANTOFIX®). An amount of the as-prepared gel was re-dispersed in distilled water under magnetic stirring to form a milky solution. An aqueous solution of hydrogen peroxide (30 wt% H_2O_2) was added dropwise to the solution under vigorous stirring for 1 h. The resultant solution was heated at 95 °C for 5 h under magnetic stirring. For the preparation of Ag and CeO_2 co-doped TiO_2 (Ag/CeO_2–TiO_2) sols, the calculated amount of $AgNO_3$ and CeO_2 were added dropwise into the TiO_2 solution during heating at 95 °C. Several hours later, the yellowish and transparent Ag/CeO_2–TiO_2 sol was obtained. The solid content of the TiO_2 in the solution was 1.0 wt%, the molar ratio of H_2O_2:TiO_2 was 4:1, and the weight ratio of Ag:CeO_2:TiO_2 was 0.5:5:100. No surfactant was needed in the preparation.

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